SWORN TRANSLATION

I, Junichi ASO, hereby declare and state that I am knowledgeable of each of the Japanese and English languages and that I made the attached translation of the attached application from the Japanese language into the English language and that I believe my attached translation to be accurate, true and correct to the best of my knowledge and ability.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

FAX RECEIVED

JUN 0 5 2003

TECHNOLOGY CENTER 2800

Date: May 27, 2003

Declarant : Junichi Aso

Junichi ASO

[Identification of Document]	Petition for Patent Application
[Reference Number]	00P00530
[Date of Submission]	November 15, 2000
[Addressee]	Commissioner, Patent Office: Kozo OIKAWA
[International Patent Classification]	H011, 21/205
[Title of the Invention]	A method for fabricating a III-V nitride film and an apparatus for fabricating the same
[Number of Claims]	8
[Inventor]	
[Address]	c/o NGK INSULATORS, LTD. 2-56, Suda-Cho, Mizuho-Ku, Nagoya City, Aichi Pref., Japan
[Name]	Tomohiko SHIBATA
[Inventor]	
[Address]	c/o NGK INSULATORS, LTD. 2-56, Suda-Cho, Mizuho-Ku, Nagoya City, Aichi Pref., Japan
[Name]	Yukinori NAKAMURA
[Inventor]	
[Address]	c/o NGK INSULATORS, LTD. 2-56, Suda-Cho, Mizuho-Ku, Nagoya City, Aichi Pref., Japan
[Name]	Mitsuhiro TANAKA
[Inventor]	
[Address]	c/o NGK INSULATORS, LTD. 2-56, Suda-Cho, Mizuho-Ku, Nagoya City, Aichi Pref., Japan
[Name]	Keiichiro ASAI
[Applicant]	
[Identification Number]	000004064
[Name]	NGK INSULATORS, LTD.
[Representative]	
[Identification Number]	100072051
[Patent Attorney]	
[Name]	Kosaku SUGIMURA
[Representative]	
[Identification Number]	100059258
[Patent Attorney]	
[Name]	Akihide SUGIMURA

[Identification of Fee]

[Deposit Account Number]	074997
[Amount of Payment]	¥21,000

[List of Attached Items]

[Identification of Item]	Specification: 1
[Identification of Item]	Drawing : 1
[Identification of Item]	Abstract : 1
[General Authorization Number]	9703804

Received from < +3152338320 > at 6/5/03 3:22:44 PM [Eastern Daylight Time]

[Identification of Document] Specification

[Title of the Invention] A method for fabricating a III-V nitride film and an apparatus for fabricating the same

[Claims]

[Claim 1] A method for fabricating a III-V nitride film, comprising the steps of:

preparing a substrate onto a susceptor in a reactor,

heating the substrate to a predetermined temperature,

coating an AlaGabIncN (a+b+c=1, a>0) film on an interior portion of a reactor which is heated to about 1000°C or over through the heating for the substrate, and

introducing a III raw material gas and a V raw material gas with a carrier gas onto the substrate prepared in the reactor, and thus, fabricating an AlxGayInzN (x+y+z=1) film by a MOCVD method.

[Claim 2] The fabricating method as defined in claim 1, wherein the AlaGabIncN film is coated on the susceptor which is heated to about 1000°C or over.

[Claim 3] The fabricating method as defined in claim 1 or 2, wherein the AlxGayInzN (x+y+z=1) film includes 50 atomic percentages or over of Al (a>0.5) for all of the group III elements.

[Claim 4] The fabricating method as defined in claim 1 or 2, wherein the AlxGayInzN (x+y+z=1) film is composed of an AlN film.

[Claim 5] The fabricating method as defined in any one of claims 1-4, wherein the AlaGabIncN (a+b+c=1, a>0) film includes 50 atomic percentages or over of Al (a>0.5) for all of the group III elements.

[Claim 6] The fabricating method as defined in any one of claims 1-4, wherein the AlaGabIncN (a+b+c=1, a>0) film is composed of an AlN film.

[Claim 7] An apparatus for fabricating a III-V nitride film by a MOCVD method, comprising:

a reactor in which the MOCVD reaction between a III raw material gas and a V material gas is generated,

a susceptor to hold a substrate thereon installed in the reactor,

a heater to heat the substrate to a predetermined temperature via the susceptor,

wherein at least one of the interior wall of the reactor and the susceptor is coated with an AlaGabIncN (a+b+c=1, a>0) film, which is heated to 1000°C or over.

[Claim 8] The fabricating apparatus as defined in claim 7, wherein the AlaGabIncN (a+b+c=1, a>0) film includes 50 atomic percentages or over of Al element (a>0.5) for all of the group III elements.

[Detailed Explanation of the Invention]

[0001]

[Technical Field of the Invention] This invention relates to a method to epitaxially grow a III-V nitride film, particularly an AlxGayInzN (x+y+z=1) film on a given substrate by a Metal Organic Chemical Vapor Deposition (MOCVD) method and an apparatus for the same method.

[0002]

[Prior Art] In opto-electronic devices such as light-emitting diodes, laser diodes or photodiodes, it is proposed that III-V nitride films having their compositions of AlxGayInzN(X+Y+Z=1) are epitaxially grown on a given substrate made of sapphire single crystal, for example. Up to now, the epitaxial growth of the AlxGayInzN film has been performed by a MOCVD (Metal Organic Chemical Vapor Deposition) method, a MOVPE (Metalorganic Vapor Phase Epitaxy) method or recently, a Hydride Vapor Phase Epitaxy (HVPE) method.

[0003] In the case of making a GaN film by a HVPE method, first of all, a substrate made of sapphire single crystal is set into a reactor in which a gallium metallic material is charged. Then, a hydrochloric acid gas is introduced into the reactor and reacted with the gallium metallic material, to generate a hydrochloric gallium gas. Then, an ammonia gas is introduced into the reactor and reacted with the hydrochloric gallium gas, to deposit and fabricate the GaN film on the substrate. The HVPE method has a higher film growth rate than a MOCVD method or a MOVPE method. For example, in the MOVPE method, a GaN film can be epitaxially grown typically at only several μm/hour, but in the HVPE method, the GaN film can be epitaxially grown typically at several hundreds μm/hour. Therefore, the HVPE method has its advantage in forming a thicker III-V nitride film.

[0004] However, a good quality AlxGayInzN film can not be provided by the

HVPE method, and the fluctuation in thickness on the same substrate may be increased. On the other hand, it takes much time to form the AlxGayInzN film by the MOVPE method, and thus, the fabrication cost of the AlxGayInzN film rises.

[0005] In the case of making an AlxGayInzN (x+y+z=1) film by a MOCVD method, a given substrate is set and held on a susceptor installed in a reactor, and is heated to a predetermined temperature by a heater. Then, a trimethyl-aluminum gas, a trimethylgallium gas, a trimethylindium gas or the like as III raw material gases are introduced with a carrier gas composed of a hydrogen gas or a nitrogen gas into the reactor. On the other hand, an ammonia gas as a V raw material gas is introduced with a carrier gas composed of a hydrogen gas or a nitrogen gas into the reactor. Then, the III raw material gases and the V raw material gas are reacted, to deposit and form the AlxGayInzN film on the substrate. As the AlxGayInzN film, an aluminum nitride film, a gallium nitride film, an indium nitride film, an aluminum-gallium nitride film, an aluminum-indium nitride film and a gallium-indium nitride film are exemplified.

[0006] In the above conventional method such as a MOCVD method, if the reaction between the III raw material gases and the V raw material gas is created on the wall surfaces of the reactor, the film-forming efficiency is degraded, and thus, the film growth rate is decreased. In the past, therefore, the raw material gases are cooled down at their introduction to the reactor, or the interior wall of the reactor are partially cooled down. On the other hand, the ammonia gas as the V raw material gas exhibits its intensive corrosion property. Therefore, the heated parts of the interior wall of the reactor may be coated by a protective layer made of SiC, p-BN, TaCx, NbNx, etc., so as to prevent the corrosion of the interior wall.

[0007] In this case, if the reaction between the III raw material gases and the V raw material gas is created on the wall surfaces of the reactor, the resulting AlpGaqInrN (p+q+r=1) compound is not deposited on the protective layer, but is drop off of the protective layer as particles. Particularly, at the highly heated parts of the protective layer, more particles composed of the AlpGaqInrN compound are created through the gas phase reaction between the III raw material gases and the V raw material gas due to the catalytic effect of the

interior wall of the reactor. In the case of using a trimethylaluminum gas as one of the III raw material gases for making an AlN film or an Al-rich AlxGayInzN (x+y+z=1, x>0.5) film, many particles are easily created due to the higher reactivity of the trimethylaluminum gas.

[0008] The resulting particles are drop off from on the protective layer, as mentioned above, and blown off by the raw material gases or the carrier gas and deposited onto a substrate. In this case, the quality of the resulting AlxGayInzN film, particularly an AlN film or an Al-rich AlxGayInzN film is deteriorated due to the deposited particles.

[0009] In addition, since the substrate on which the AlxGayInzN film is formed is heated through a susceptor which is initially heated with a heater, the surface temperature of the susceptor is raised to higher temperature of 1000°C or over than the substrate temperature, so that more particles composed of the AlpGaqInrN compound are easily created on the surface of the susceptor. Then, the created particles are deposited on the substrate, and thus, the quality of the resulting AlxGayInzN film is deteriorated.

[0010] Fig. 1A is a conceptual view showing the film quality of an AlN film formed on a substrate made of a 3-inch wafer using a new susceptor.
The quality deterioration of the AlN film is designated by the hatched region at the periphery of the substrate. As shown in Fig. 1A, the quality of the AlN film is remarkably deteriorated by using the new susceptor due to the particle deposition. Particularly, a new susceptor is susceptible to the deposition of particles of aluminum nitride, resulting in the deterioration of the resultant AlN film.

[0011] Moreover, when using such a large substrate as a 3-inch wafer so as to reduce the fabrication cost, the quality deterioration of the resulting AlxGayInzN film becomes remarkable due to more particles at the periphery of the substrate.

[0012]

[Task to be Solved by the Invention] It is an object of the present invention to work out the above conventional problems, and thus, to provide a method and an apparatus for epitaxially growing a good quality AlxGayInzN film (x+y+z=1) by a MOCVD method.

[0013] In order to achieve the above object, this invention relates to a method for fabricating a III-V nitride film, comprising the steps of:

preparing a substrate onto a susceptor in a reactor,

heating the substrate to a predetermined temperature,

coating an AlaGabIncN (a+b+c=1, a>0) film on an interior portion of a reactor which is heated to about 1000°C or over through the heating for the substrate, and

introducing a III raw material gas and a V raw material gas with a carrier gas onto the substrate prepared in the reactor, and thus, fabricating an AlxGayInzN (x+y+z=1) film by a MOCVD method.

[0014] The coated AlaGabIncN (a+b+c=1, a>0) film includes unavoidable elements such as oxygen, silicon, magnesium and another element containing in the interior wall of the reactor by several %. Moreover, the AlaGabIncN film is not required to have a uniform composition over the total thickness, but may have a continuously or stepwisely inclined composition or an multi-layered structure composed of plural films having their respective different compositions.

[0015] In a preferred embodiment of the present invention, the AlaGabIncN (a+b+c=1, a>0) film is coated on the susceptor to hold the substrate. In the case of making a III-V nitride film by a MOCVD method, as mentioned above, the substrate is heated to around 1000°C, and thus, the surface of the susceptor is heated to 1000°C or over. Therefore, more particles composed of the AlpGaqInrN (p+q+r=1) compound is likely to be created on the susceptor. However, if the susceptor is coated with the AlaGabIncN film, the AlpGaqInrN compound is deposited directly on the susceptor, and thus, the particles are not created and deposited on the substrate. As a result, the resulting AlxGayInzN film can have its desirable quality.

[0016] The fabricating method of the present invention may be preferably usable in forming an Al-rich AlxGayInzN (x+y+z=1, x>0.5) film or an AlN film by introducing into the reactor much amount of trimethylaluminum gas with an ammonia gas.

[0017] In the fabricating method of the present invention, the substrate may be made of Al_2O_3, SiC, $NdGaO_3$, $LiGaO_3$, AlGaInN, ZnO, MgO or $MgAl_2O_4$. Moreover, the substrate may be composed of an epitaxial substrate composed of a base made of such a single crystal and a given epitaxial film formed on the base.

[0018] This invention also relates to an apparatus for fabricating a III-V

nitride film by a MOCVD method, comprising:

a reactor in which the MOCVD reaction between a III raw material gas and a V material gas is generated,

a susceptor to hold a substrate thereon installed in the reactor,

a heater to heat the substrate to a predetermined temperature via the susceptor,

wherein at least one of the interior wall of the reactor and the susceptor is coated with an AlaGabIncN (a+b+c=1, a>0) film, which is heated to 1000°C or over.

[0019] In a preferred embodiment of the present invention, the AlaGabIncN (a+b+c=1, a>0) film is coated on the susceptor to hold the substrate. It is desired that the AlaGabIncN (a+b+c=1, a>0) film includes 50 atomic percentages or over of Al (a>0.5) for all of the group III elements, and particularly, is composed of an AlN film.

[0020]

[Mode for Carrying Out the Invention] Fig. 2 is a cross sectional view diagrammatically showing the structure of a fabricating apparatus for a III-V nitride film according to the present invention. In Fig. 2, the fabricating apparatus includes a reactor 11 made of quartz, a susceptor 13 at the center in the lower side of the reactor and a heater 14 under the susceptor 13. A substrate 12 made of, e.g., sapphire single crystal is set horizontally on the susceptor and heated to a predetermined temperature with the heater. Although, in Fig. 2, the substrate is held on the upper surface of the susceptor, it may be held on the lower surface.

[0021] At the right side of the reactor 11 are provided gas inlets 15-17 to introduce raw material gases with a carrier gas. In the case of making an AlN film, for example, a trimethylaluminum gas is introduced with a hydrogen carrier gas from the first gas inlet 15, and an ammonia gas is introduced from the second gas inlet 16. Then, a carrier gas composed of a hydrogen gas or a nitrogen gas is introduced from the third gas inlet 17. The introduced trimethylaluminum gas and the introduced ammonia gas are also introduced into the center region of the reactor through separated guiding tubes 18 and 19, respectively. In this case, the raw material gases are effectively supplied onto the substrate 12, and not

supplied in the remote region from the substrate 12. The introduced raw material gases are consumed by a MOCVD reaction on the substrate, and the remaining raw material gases are discharged from gas outlet 20 provided at the left side of the reactor 11.

[0022] In the case of making the AlN film, the substrate 12 is heated to around 1000°C, for example by the heater 14. In this case, the surface temperature of the susceptor 13 is raised to 1000°C or over. Therefore, the raw material gases are likely to be chemically reacted on the susceptor, to create particles composed of the AlNx compound. The particles are blown off by the raw material gases and the carrier gases and deposited on the substrate 12, to deteriorate the quality of the AlxGayInzN film, that is, the AlN film. Therefore, as shown in Fig. 3, for example, another AlN film 21 is coated in a thickness of 1 μm on the top surface and the side surfaces of the susceptor 13. In this case, the AlNx compound is deposited on the coated AlN film 21, and thus, particles composed of the AlNx compound are not almost created. As a result, the resulting AlN film is not affected by the particles, and can have its desirable quality over the almost entire main surface of the substrate including the periphery thereof, as shown in Fig. 1B. Accordingly, the fabricating total cost of the AlN film can be reduced.

[0023] Although the present invention was described in detail with reference to the above example, this invention is not limited to the above disclosure and every kind of variation and modification may be made without departing from the scope of the present invention. For example, the AlN film 21 may be coated on the interior walls of the reactor and/or another instrument installed in the reactor which are heated to higher temperature of 1000°C or over, instead of coating over the susceptor 13. Then, besides the AlN film, another AlaGabIncN (a+b+c=1, a>0) film or AlaGabIncN (a+b+c=1, a>0.5) film may be coated. The substrate 12 may be made of, instead of the sapphire single crystal, SiC, $NdGaO_3$, $LiGaO_3$, AlGaInN, ZnO, MgO or $MgAl_2O_4$. Moreover, the substrate 12 may be constructed of an epitaxial substrate composed of a base made of such a single crystal and a given epitaxial film formed on the base. The AlaGabIncN films may be formed by means of any film forming method.

[0024] The configuration of the susceptor may be changed and modified.

The substrate 12 may be set in a grooved portion 13a formed at the plane main surface of the susceptor 13 as shown in Fig. 4, instead of being set directly on the plane main surface of the susceptor 13. In this case, for not disturbing the raw material gas flow on the substrate 12, it is desired that the grooved portion is formed so that the surface level of the substrate 12 set into the grooved portion can be the same as the surface level of the coated AlN film 21. Moreover, the AlN film 21 may not be coated at the connection between the substrate 12 and the susceptor 13 and/or the side surfaces of the susceptor 13 to which the raw material gases are not directly contacted.

[0025] As mentioned above, according to the fabricating method and the fabricating apparatus for a III-V nitride film of the present invention, an AlaGabIncN (a+b+c=1, a>0) film, particularly an Al-rich AlaGabIncN (a+b+c=1, a>0.5) film, more particularly an Al film is coated on an interior portion of the reactor which is heated to 1000°C or over. Therefore, the AlpGaqInrN (p+q+r=1) compound generated through the reaction of the raw material gases is deposited on the coated film, and thus, particles made of the AlpGaqInrN compound are not almost created. As a result, the resulting AlxGayInzN film is not affected by the particles, and can have its desirable quality. In addition, the interior portion including the interior wall of the reactor is not almost corroded by an ammonia gas as a V raw material gas because the coated film functions as an anti-corrosive film, so that the durability of the whole fabricating apparatus can be developed.

[Brief Description of the Drawings]

[Fig. 1] It is a conceptual view showing the film quality of an AlN film formed on a substrate made of a 3-inch wafer, according to the present invention.

[Fig. 2] It is a cross sectional view diagrammatically showing the structure of a fabricating apparatus for a III-V nitride film according to the present invention.

[Fig. 3] It is a cross sectional view showing the susceptor of the fabricating apparatus.

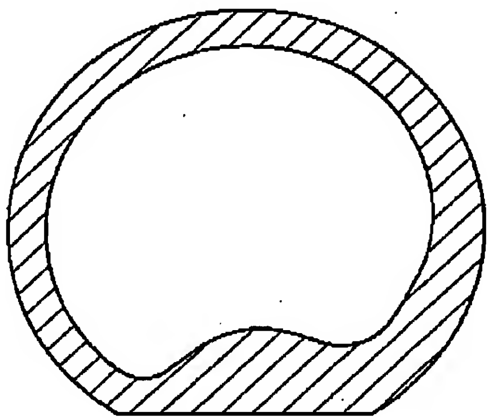
[Fig. 4] It is a cross sectional view showing the susceptor of another fabricating apparatus according to the present invention.

[Description of the Reference Numerals]

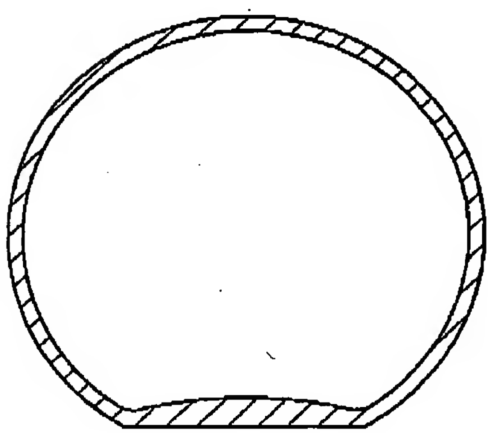
11: reactor, 12: substrate, 13: susceptor, 14: heater, 15, 16, 17: gas inlet, 18, 19: guiding tube, 20: gas outlet, 21: AlN film

【書類名】 図面
[Identification of Document] Drawing
【図１】
[Fig. 1]

A

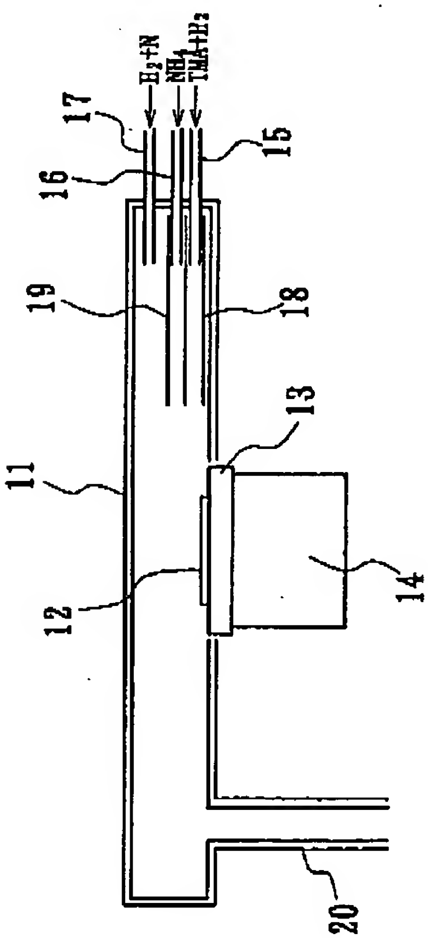


B



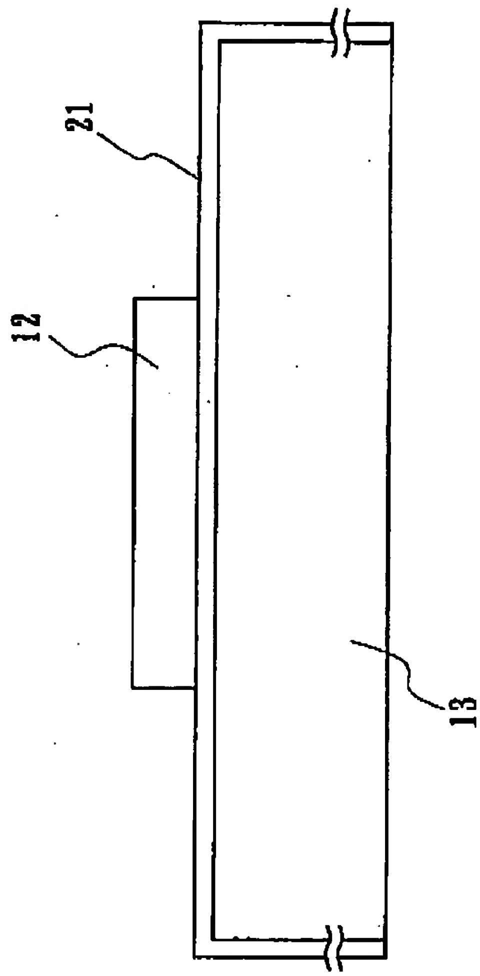
【図２】

[Fig. 2]

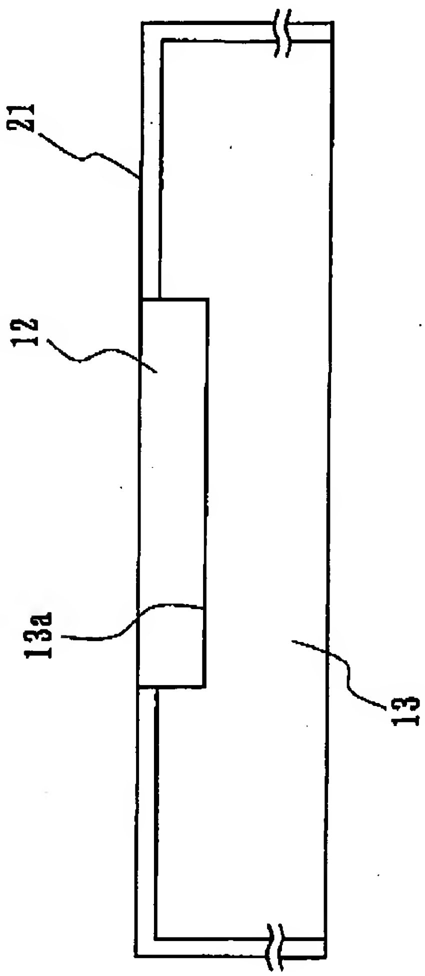


【図３】

[Fig. 3]



【図４】
[Fig. 4]



[Identification of Document] Abstract

[Abstract]

[Object] It is an object to provide a method and an apparatus for epitaxially growing a good quality AlxGayInzN film (x+y+z=1) by a MOCVD method.

[Solving Means] The substrate 12 is set on the susceptor 13 installed in the reactor 11. Then, the trimethylaluminum gas and the ammonia gas with the carrier gas are introduced into the reactor 11 through the gas inlets provided at the side thereof, and are chemically reacted to form the intended AlN film on the substrate 12. Since the surface of the susceptor 13 is heated to at least 1000°C, it is coated with the AlN film 21 in advance so that the particles from the deposited debris are not created and deposited on the substrate 12. As a result, the quality of the intended AlN film is not deteriorated.

[Selected Figure] Fig. 2

拒絶理由通知書

特許出願の番号	特願２０００－３４７７３５
起案日	平成１５年　７月　７日
特許庁審査官	加藤　浩一　　　　　８６１７　４Ｅ００
特許出願人代理人	杉村　興作（外　１名）　様
適用条文	第２９条第１項、第２９条第２項、第２９条の２

この出願は、次の理由によって拒絶をすべきものである。これについて意見があれば、この通知書の発送の日から６０日以内に意見書を提出して下さい。

理　由

Ｉ．この出願の下記の請求項に係る発明は、その出願前に日本国内又は外国において、頒布された下記の刊行物に記載された発明又は電気通信回線を通じて公衆に利用可能となった発明であるから、特許法第２９条第１項第３号に該当し、特許を受けることができない。

II．この出願の下記の請求項に係る発明は、その出願前日本国内又は外国において頒布された下記の刊行物に記載された発明又は電気通信回線を通じて公衆に利用可能となった発明に基いて、その出願前にその発明の属する技術の分野における通常の知識を有する者が容易に発明をすることができたものであるから、特許法第２９条第２項の規定により特許を受けることができない。

記　　　（引用文献等については引用文献等一覧参照）

・理由Ｉ、IIについて
・請求項　　　１－８
・引用文献等　１－２

・備考
引例１の請求項１、請求項３、引例２の請求項６等を参照されたい。

III.この出願の下記の請求項に係る発明は、その出願の日前の特許出願であって、その出願後に出願公開がされた下記の特許出願の願書に最初に添付された明細書又は図面に記載された発明と同一であり、しかも、この出願の発明者がその出願前の特許出願に係る上記の発明をした者と同一ではなく、またこの出願の時に

おいて、その出願人が上記特許出願の出願人と同一でもないので、特許法第２９条の２の規定により、特許を受けることができない。

記　　（引用文献等については引用文献等一覧参照）

・請求項　　１－８
・引用文献等　３

・備考
先願明細書３の請求項４、請求項７、請求項１３等を参照されたい。

引 用 文 献 等 一 覧

1.特開平１０－２８４４２５号公報
2.特開平１０－０７４７０５号公報
3.特願２０００－１６１５５０号（特開２００１－３４５２６８号公報）

－－－

先行技術文献調査結果の記録

・調査した分野　　ＩＰＣ第７版　Ｈ０１Ｌ２１／２０５

この先行技術文献調査結果の記録は、拒絶理由を構成するものではない。

この拒絶理由通知の内容に関するお問い合わせ、または面接のご希望がございましたら下記までご連絡下さい。

特許審査第三部金属加工　加藤　浩一
TEL. 03(3581)1101 内線3425
FAX. 03(3580-6905)